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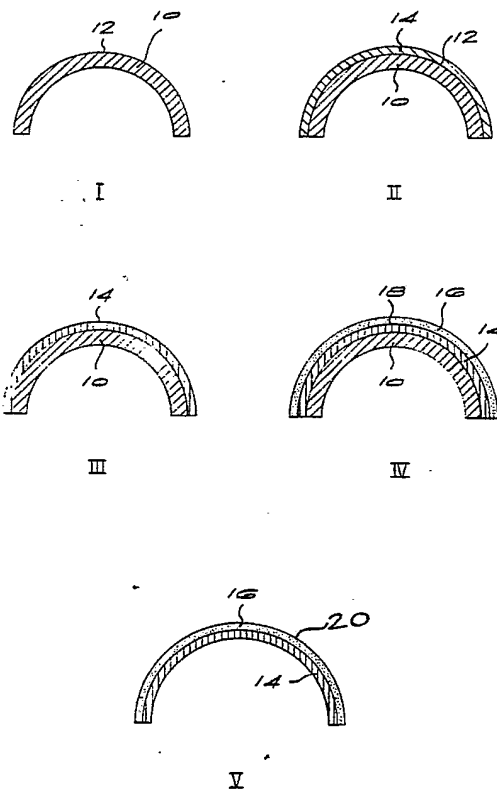
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54 **Diamond growth.**

57 A method of producing a diamond or diamond-like film (16) having a desired profile which involves providing a solid carbon substrate (10) having a surface (12) shaped to the desired profile, creating a thin carbide layer (14) on the profiled surface (12), growing a diamond or diamond-like film (16) on the carbide layer (14), removing the carbon substrate (10) and optionally also removing the carbide layer (14).



**EP 0 400 947 A1**

## DIAMOND GROWTH

### BACKGROUND OF THE INVENTION

This invention relates to diamond growth.

Various methods have been proposed and tried for growing diamond on diamond seed crystals by chemical vapour deposition (CVD) using gaseous carbon compounds such as hydrocarbons or carbon monoxide. A gaseous compound can be produced from a liquid carbon compound such as an alcohol or acetone. The gaseous carbon compounds may be decomposed by various methods including the use of heat and radio frequency (RE) energy, and also by means of microwave energy.

Examples of patents directed to methods of growing diamonds on substrates are United States Patents Nos. 4,434,188 and 4,734,339 and European Patent Publications Nos. 288065, 327110, 305903 and 286306.

European Patent Publication No. 0348026 describes a method of growing crystalline diamond on a substrate which includes the steps of providing a surface of a suitable nitride, placing the substrate on the nitride surface, creating an atmosphere of a gaseous carbon compound around the substrate, bringing the temperature of the nitride surface and the substrate to at least 60°C, and subjecting the gaseous carbon compound to microwave energy suitable to cause the compound to decompose and produce carbon which deposits on the substrate and forms crystalline diamond thereon. The nitride is typically silicon nitride and the frequency of the microwave is typically in the range 200 MHz to 90 GHz.

### SUMMARY OF THE INVENTION

According to the present invention, a method of producing a diamond or diamond-like film of a desired profile includes the steps of providing a solid substrate having a surface shaped to the desired profile, producing a thin carbide layer on the profiled surface, growing a diamond or diamond-like film on the carbide layer, removing the substrate and optionally removing the carbide layer.

### DESCRIPTION OF THE DRAWINGS

The drawing illustrates schematically the various steps of an embodiment of the method of the invention.

### DESCRIPTION OF EMBODIMENTS

The solid substrate will be made of a material which can easily be formed into a desired shape and which is inert to microwave or like energy. Examples of suitable materials are silicon nitride and, preferably, carbon, e.g. graphite.

The carbide layer will be thin and generally less than 20 microns in thickness. Preferably, the thickness of the layer will be less than 5 microns. The layer will thus follow closely the profile of the shaped surface.

The carbide layer will preferably be a carbide of a high melting metal such as titanium, hafnium, zirconium, molybdenum, tantalum or the like. This layer is preferably formed by first depositing a layer of the metal, for example by evaporation, on the shaped surface and thereafter exposing the metal layer to an atmosphere containing carbon plasma to convert the metal to a carbide.

The diamond or diamond-like film which is grown on the carbide layer by chemical vapour deposition such as that described in European Patent Publication No. 0348026. The layer which will generally be polycrystalline in nature.

The solid substrate and/or carbide layer may be removed by machining, milling or etching, or a combination of such methods.

The invention produces a profiled diamond or diamond-like film which has a large surface area and which is thin. The thickness of the diamond or diamond-like film will generally be less than 100 microns. The film will, of course, have a major surface and this surface will generally have an area of at least 10mm<sup>2</sup>. The film can be any one of a variety of shapes such as curved, convoluted, and the like.

The diamond or diamond-like film may be left bonded to the thin carbide layer. In this form it is essentially free standing. Alternatively, the carbide layer may be removed producing a diamond or diamond-like film which is completely free standing.

An embodiment of the invention will now be described with reference to the accompanying drawings. Referring to these drawings, there is shown in stage I a graphite substrate 10 having a curved, semi-circular upper surface 12. In stage II a layer 14 of titanium is evaporated on to the curved surface 12. Typically, this layer 14 will have a thickness of less than 5 microns. Because of the thinness of this layer, it follows closely the profile of the surface 12.

The titanium layer is converted to titanium carbide in stage III. This is achieved by exposing the

coated graphite substrate 10 to microwave energy in the presence of a mixture of methane and hydrogen, the methane constituting 15% to 80%, preferably 50%, by volume of the mixture. The methane decomposes to produce carbon plasma which reacts with the titanium to form titanium carbide.

Then, in stage IV a diamond film 16 is grown on the upper curved surface 18 of the titanium carbide layer 14 by any known method of producing a diamond film on a substrate by chemical vapour deposition.

In stage V, the graphite substrate is machined or etched away leaving a diamond film 16 on a thin titanium carbide backing 14. This diamond film is essentially free-standing. The titanium carbide backing 14 can be removed, for example by milling, plasma etching or chemical digestion to produce a diamond film which is completely free-standing.

The diamond film will typically have a thickness of less than 100 microns and its upper surface 20 will typically have an area of at least 10mm<sup>2</sup>.

9. A method according to any one of the preceding claims wherein the diamond or diamond-like film (16) is grown on the carbide layer (14) by chemical vapour deposition.

## Claims

1. A method of producing a diamond or diamond-like film (16) having a desired profile includes the steps of providing a solid substrate (10) having a surface (12) shaped to the desired profile, producing a thin carbide layer (14) on the profiled surface (12), growing a diamond or diamond-like film (16) on the carbide layer (14) and removing the substrate.

2. A method according to claim 1 wherein the carbide layer (14) is also removed.

3. A method according to claim 1 or claim 2 wherein the substrate (10) is a solid carbon substrate.

4. A method according to claim 3 wherein the substrate (10) is a graphite substrate.

5. A method according to any one of the preceding claims wherein the carbide is a carbide of a high melting metal selected from titanium, hafnium, zirconium, molybdenum and tantalum.

6. A method according to any one of the preceding claims wherein the carbide layer (14) has a thickness of less than 20 microns.

7. A method according to any one of claims 1 to 4 wherein the carbide layer (14) has a thickness of less than 5 microns.

8. A method according to any one of the preceding claims wherein the solid substrate (10) and/or carbide layer (14) are removed by a method selected from machining, milling, etching and a combination thereof.

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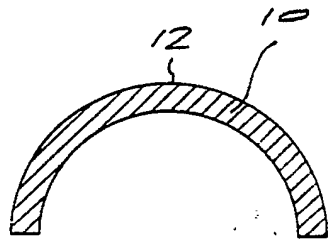
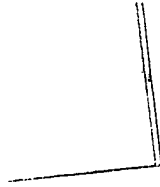
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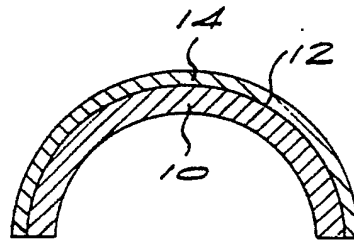
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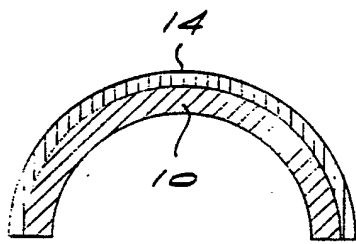
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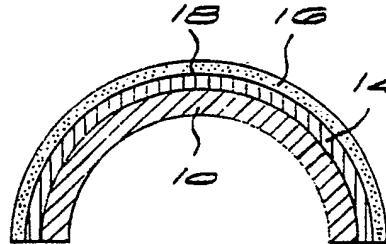
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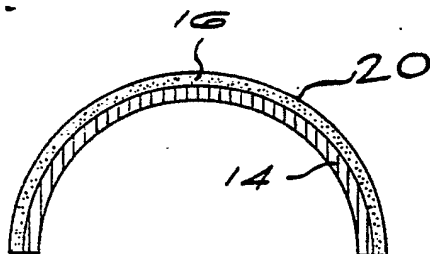
II



III



IV



V



European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number

EP 90 30 5794

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	US-A-4 332 751 (G.W. BRASSELL et al.) * Abstract; column 2, lines 39-57 * ---	1,9	C 23 C 16/00 C 23 C 16/26
A	NUCLEAR INSTRUMENTS AND METHODS, vol. 119, 1974, pages 321-322, North-Holland Publishing Co., Amsterdam, NL; R. KELLER et al.: "The preparation of self-supporting <sup>13</sup> C-foils using enriched methane gas" * Whole document * ---	1,9	
A	GB-A-2 099 806 (THE SECRETARY OF STATE FOR DEFENCE) * Abstract * ---	1	
A	EP-A-0 076 731 (UNION CARBIDE CORP.) ---		
A	US-A-4 550 014 (R.J. BAUGHMAN et al.) ---		
A	US-A-4 271 235 (L. HILL et al.) -----		
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 23 C C 30 B
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 10-09-1990	Examiner JOFFREAU P.O.
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document  T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... & : member of the same patent family, corresponding document			